California Environmental Protection Agency

Air Resources Board

Special Analysis Section
Northern Laboratory Branch
Monitoring and Laboratory Division

MLD SOP SAS07

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF EXEMPT AND NON-EXEMPT COMPOUNDS GENERALLY FOUND IN CONSUMER PRODUCTS BY GAS CHROMATOGRAPHY-FID

January 18, 2005, Revision 1.6

DISCLAIMER: Mention of any trade name or commercial product in Method 310 and associated Standard Operating Procedures does not constitute endorsement or recommendation of this product by the Air Resources Board. Specific brand names and instrument descriptions listed in the Standard Operating Procedures are equipment used by the ARB laboratory. Any functionally equivalent instrumentation can be used.

1 INTRODUCTION

In the analysis of volatile organic compounds (VOCs) in consumer products, the percent total volatile content is determined by MLD SOP SAS01. From this total is subtracted that portion of volatile material not classified as VOC, e.g. water and ammonium. Several compounds have been identified as less reactive and therefore exempt from the VOC definition and are subtracted from the total volatile content. To characterize the volatile content present in a consumer product, this procedure looks at several compounds both exempt and non-exempt.

Appendix A describes the determination of the exempt compounds: ethanol (AP/DO only), acetone, methyl acetate and perchloroethylene. Additional analytes are methanol, isopropanol, 1-propanol, isobutanol, and limonene.

Appendix B describes the determination of the exempt compounds: dichloromethane and 1,1,1-trichloroethane. Additional analytes are ethyl acetate, toluene, ethyl benzene, xylenes, and pinene.

Appendix C describes the determination of the exempt compounds: hexamethyldisiloxane, hexamethylcyclotrisiloxane, octamethyltrisiloxane, octamethylcyclotetrasiloxane, decamethyltetrasiloxane, and decamethylcyclopentasiloxane. One additional analyte seen is benzyl alcohol.

Appendix D describes the determination of the exempt compound p-chlorobenzotrifluoride. Additional analytes are methyl ethyl ketone, propylene glycol methyl ether acetate (PM acetate), and 2-butoxyethanol.

Appendix E describes the determination of the exempt compounds: ethylene glycol, propylene glycol, 2-(2-ethoxyethoxy)ethanol (Carbitol), glycerol, 2-(2-butoxyethoxy)ethanol (Butyl Carbitol), and triethylene glycol.

2 SUMMARY OF METHOD

The samples of consumer products are prepared as 1:10 wt. / volume dilutions in 1-methoxy-2-propanol (MPA). After thorough mixing, the solution may require filtering to remove insoluble material. If under special circumstances another solvent is required, then the standards, control, checks and trip sample are to be made with the same solvent and analyzed with those samples.

The diluted sample is then analyzed on a gas chromatograph equipped with a flame ionization detector. The data is reported as weight fraction of analyte in the product.

3 INTERFERENCES AND LIMITATIONS

With the increase in the number of compounds being identified, overlap of the retention times may start to occur. Care must be taken to make certain of the identity of the compounds, if possible through gas-chromatography-mass spectrometery.

4 INSTRUMENTATION AND EQUIPMENT

4.1 Gas Chromatograph (GC) configured with a Flame Ionization Detector (FID)

4.1.1 GC Column: J & W DB-624, 30 m x 0.32 mm I.D. with 1.8 µm film

4.1.2 GC Parameters are as follows:

Oven Conditions	
Initial temperature:	35℃
Initial time:	5.0 min
Rate:	10℃/min
Final temperature:	200℃
Final time:	1.0 min
Run time:	22.5 min
Oven equilibration:	0.3 min
Injector temperature:	250℃
Detector temperature:	250℃
Carrier gas (He):	10 psi (26 cm/sec)
DET B FID:	ON
EPP B:	9.5 psi @ 35^{o}C
Split Ratio:	100 mL/min

4.2 Volumetric Flasks:

4.2.1 10 mL,

4.2.2 500 mL

4.3 Rainin Pipettors:

4.3.1 250 µL with tips,

4.3.2 1.0 mL with tips,

4.3.3 2.5 mL with tips

4.4 Vials:

4.4.1 20 mL, for standards,

4.4.2 8 mL with PTFE-lined cap, for standards and dilutions,

4.4.3 2 mL with caps, for GC analysis

4.5 Analytical Balance:

4.5.1 Sartorious ME215S,

4.5.2 Sartorious MC1

4.6 Vortex Mixer, variable speed

4.7 Homogenizer

5 REAGENTS

5.1 1-Methoxy-2-propanol (MPA), 98%

5.2 Analytes, spectrophotometric grade - see specific appendix for listing.

5.3 Stock Standards: The 80 mg/mL stock standard is prepared gravimetrically. Analytes are specified in each of the appendices.

5.4 Control/Check Stock Solution: A control/check stock solution is prepared using a specified analyte in MPA, for each of the appendices covered in this SOP. The analyte is weighed in the preparation of the stock, so the concentration is in g/mL.

5.5 Trip Sample Stock Solution (Appendix A analysis only): A trip sample stock solution is prepared by weighing 300 g of water and 50 g each of sodium chloride, acetone, methanol, and ethanol into a 500 mL volumetric flask and bringing to volume with MPA.

5.6 Helium, grade 5

5.7 Air, compressed, ultra high purity

5.8 Hydrogen Generator:

5.8.1 Whatman, model 75-32 or equivalent,

5.8.2 20 – 25 psi output

6 PROCEDURE

6.1 Instrument Preparation:

6.1.1 Turn on the main valve for the air cylinder; verify cylinder pressure is above 500 psi.

6.1.2 Verify helium cylinder pressure is above 500 psi.

6.1.3 Check that the water level in hydrogen generator is above the refill line.

6.1.4 Press the FID igniter on the front of the GC:

6.1.4.1 Confirm ignition by looking for water vapor on a mirror held up to the FID outlet.

6.2 Analysis Preparation:

6.2.1 Solvent Blank: Prepare solvent blank by filling a GC vial with the same MPA used to make the dilutions in steps 6.2.2 – 6.2.4. Cap the vial.

6.2.2 Calibration Standards: Prepare the five calibration standards in 10 mL volumetric flasks as follows:

Concentration	Volume of Stock Standard
1.0 mg/mL	0.125 mL
10 mg/mL	1.25
20 mg/mL	2.50
40 mg/mL	5.0
80 mg/mL	----

Bring to volume with MPA, mix thoroughly and place in dilution vials.

6.2.3 Transfer an aliquot of each standard into a GC vial and cap.

6.2.4 Control/Check: Prepare the control/check by diluting 1.0 mL of the control/check stock standard to 10 mL with MPA. The control is analyzed after the calibration. The check is run after every ten samples and at the end of the run.

6.2.5 Trip Sample (Appendix A analysis only): Prepare the trip sample by diluting 1.0 mL of the trip stock standard to 10 mL with MPA. The trip sample is run after the control.

6.2.6 Sample: A 1.0 mL aliquot of the consumer product sample is weighed into a 10 mL volumetric flask. After the weight is recorded, the aliquot is diluted to 10 mL with MPA.

6.2.7 Transfer an aliquot of each control/check, trip and sample into appropriately labeled GC vials and cap.

6.3 Sample Analysis:

6.3.1 Place vials in the autosampler in the following order: MPA blank, calibration standards, control/check, trip sample, and diluted samples. The check standard is run every tenth sample and at the end of the run. Additional blanks between standards and samples maybe used if carryover is suspected.

6.3.2 Perform Sample Analysis: See appropriate appendices based on analytes of interest. Calculate the value for each analyte found by dividing the amount from the report (mg/mL) by the sample dilution weight (see Section 8).

7 QUALITY CONTROL

7.1 An MPA solvent blank must be analyzed for each batch of samples. The analyte concentration in the blank must be less than 0.1% wt./volume. An MPA blank is run before the control and each check to prevent carry over from the previous sample.

7.2 The correlation coefficient for compounds present in the calibration must be greater than 0.98. If the calibration fails, the sequence is stopped and corrective action is implemented. Corrective action may include replacing the inlet liner, reanalyzing the calibration curve or making up a new dilution of the calibration curve and then reanalyzing.

7.3 A control sample is run after the calibration. The control must fall within the control limits. If the control is not within the control limits, it may be necessary to recalibrate and rerun the sequence.

7.4 A check sample is run after every ten samples and at the end of the run. The check must fall within the control limits. If one of the checks is out of the control limits, re-run the check and any samples that follow until the next check.

7.5 The trip sample contains 10% ethanol, 10% methanol and 10% acetone. The recovery for the trip sample should be within the error of the method (± 3%).

7.6 LIMS assigns at least one duplicate sample for every sample set.

7.7 Each limit of detection (LOD) is determined annually.

8 CALCULATIONS

The weight fraction of analyte in the product is calculated as follows:

$$\text{Weight Fraction of Analyte} = \left(\frac{\text{analyte (mg/mL)}}{\text{sample dilution (g)}} \right) \times 10^{-2}$$

Appendix A

ANALYSIS FOR ACETONE/ALCOHOL

Calibration Standard Stock: The stock standard consists of the following compounds, which are placed in a 500 mL volumetric flask and prepared, as follows:

Analyte	Amount (g)
Methanol	38
Ethanol	44
Acetone	40
Isopropanol	38
1-Propanol	40
Isobutane	40
Perchloroethylene	40
Limonene	40

Bring to volume with MPA and mix. Aliquot the solution into 20 mL screw-capped vials. Date, label and store the vials in the Standards refrigerator.

Control/Check Stock: The control/check stock is a 25% acetone solution prepared by weighing 50 g each of acetone and water into a 200 mL volumetric flask. Bring to volume with MPA and mix. Aliquot the control/check stock in individual 8 mL screw-capped vials. Date, label and store the vials in the Standards refrigerator.

1 Load Method.

1.1 Under ***Method***, select ***Load Method***.

1.2 Click on ***acetone.m***; then ***OK***.

2 Load Sequence.

2.1 Under ***Sequence***, select ***Load Sequence***.

2.2 Click on ***ACETONE.S***; then ***OK***.

3 Modifying Sequence.

3.1 Under ***Sequence***, select ***Sequence Parameters*** to create a subdirectory for the data.

3.2 Input sequence parameters.

3.2.1 **Operator Name** – enter your three initials.

3.2.2 **Data Files** – select ***Auto***.

3.2.3 **Subdirectory** – enter the two decimal month, day, year (December 5, 2007 would be entered as ***120507***); a letter can be put at the end to distinguish specific analyses run on the same day.

3.2.4 **Part of method to run** – select ***According to Runtime Checklist***.

3.2.5 **Sequence Comment** – enter set sample numbers, and any other pertinent information.

3.3 Click on ***OK***.

4 Sequence Table.

4.1 Under ***Sequence***, select ***Sequence Table***.

4.2 Select the ***Back*** injector.

4.3 Input sequence information.

4.3.1 **Sample Name** – enter the following, in order: MPA blank, the five standards, MPA blank, control, trip sample and the samples.

4.3.1.1 An entry for MPA blank followed by a check is made after every ten samples and again at the end of the run.

4.3.2 **Method Name** – select ***ACETONE*** from the pull-down menu.

4.3.3 **Vial** – vial number corresponds to the position of the vial on the autosampler tray.

4.3.3.1 Each entry for the blank will reference the same blank vial.

4.3.3.2 Each entry for check will reference the control vial.

4.3.4 **Inj/Vial** – enter 1, for all samples.

4.3.5 **Sample Type** – select ***Sample*** from the pull down menu for all lines except for the five calibration lines; for these select ***Calibration***.

4.3.6 For the calibration lines only:

4.3.6.1 **Cal Level** – enter 1,2,3,4,5, respectively.

4.3.6.2 **Update RF** – select ***Replace*** from the pull down menu.

4.3.6.3 **Update RT** – select ***Average*** from the pull down menu.

4.4 Click on ***OK***.

5 Save Sequence.

5.1 Under ***Sequence***, select ***Save Sequence***.

6 Print Sequence.

6.1 Under ***Sequence***, select ***Print Sequence***.

6.2 Check ***Method and Injection Info Part***; then click on ***Print***.

6.3 Verify that the sequence matches the vial placement on the autosampler.

6.4 Place sequence printout in the sequence binder.

7 Starting Sequence.

7.1 Under ***Run Control***, select ***Run Sequence***.

8 During the Sequence Run.

8.1 Print out the calibration curve for the standards.

8.1.1 Under ***View***, select ***Data Analysis***.

8.1.2 Under ***File***, select ***Print***.

8.1.3 Select ***Calib Table + Curves***.

8.2 Verify the correlation coefficient for each compound present in the calibration is greater than 0.98.

8.2.1 If the calibration fails, the sequence is stopped and corrective action is implemented.

8.3 Verify the trip sample recoveries.

8.4 Verify control and check values are within control limits.

9 After Sequence Completion.

9.1 Review the chromatograms. Verify the MPA peak is of a consistent area, throughout the entire sequence. If a sample was misinjected, the problem will most likely show up in the MPA peak.

10 Note any problems in the lab instrument notebook.

Appendix B

ANALYSIS FOR DICHLOROMETHANE AND TRICHLOROETHANE

Calibration Standard Stock: The stock standard consists of the following compounds, which are placed in a 500 mL volumetric flask and prepared, as follows:

Analyte	Amount (g)
Dichloromethane	40
Ethyl acetate	40
1,1,1-Trichloroethane	40
Toluene	40
Ethyl benzene	40
m-Xylene	40
o-Xylene	40
Pinene	40

Bring to volume with MPA and mix. Aliquot the solution into 20 mL screw-capped vials. Date, label and store the vials in the Standards refrigerator.

Control/Check Stock: The control/check stock is a 25% dichloromethane (DCM) solution prepared by weighing 25 g of DCM into a 100 mL volumetric flask. Bring to volume with MPA and mix. The DCM is weighed out in the preparation of the stock, so the concentration is already g/mL. Store the control/check stock in individual 8 mL screw-capped vials in the Standards refrigerator.

1 Load Method.

1.1 Under ***Method***, select ***Load Method***.

1.2 Click on ***dcm.m***; then ***OK***.

2 Load Sequence.

2.1 Under ***Sequence***, select ***Load Sequence***.

2.2 Click on ***DCM.S***; then ***OK***.

3 Modifying Sequence.

3.1 Under ***Sequence***, select ***Sequence Parameters*** to create a subdirectory for the data.

3.2 Input sequence parameters.

3.2.1 **Operator Name** – enter your three initials.

3.2.2 **Data Files** – select ***Auto***.

3.2.3 **Subdirectory** – enter the two decimal month, day, year (December 5, 2007 would be entered as ***120507***); a letter can be put at the end to distinguish specific analyses run on the same day.

3.2.4 **Part of method to run** – select ***According to Runtime Checklist***.

3.2.5 **Sequence Comment** – enter set sample numbers, and any other pertinent information.

3.3 Click on ***OK***.

4 Sequence Table.

4.1 Under ***Sequence***, select ***Sequence Table***.

4.2 Select the ***Back*** injector.

4.3 Input sequence information.

4.3.1 **Sample Name** – enter the following, in order: MPA blank, the five standards, MPA blank, control, and the samples.

4.3.1.1 An entry for MPA blank followed by a check is made after every ten samples and again at the end of the run.

4.3.2 **Method Name** – select ***DCM*** from the pull-down menu.

4.3.3 **Vial** – vial number corresponds to the position of the vial on the autosampler tray.

4.3.3.1 Each entry for the blank will reference the same blank vial.

4.3.3.2 Each entry for check will reference the control vial.

4.3.4 **Inj/Vial** – “1”.

4.3.5 **Sample Type** – select ***Sample*** from the pull down menu for all lines except

for the five calibration lines; for these select ***Calibration***.

4.3.6 For the calibration lines only:

4.3.6.1 **Cal Level** – enter 1,2,3,4,5, respectively.

4.3.6.2 **Update RF** – select ***Replace*** from the pull down menu.

4.3.6.3 **Update RT** – select ***Average*** from the pull down menu.

4.4 Click on ***OK***.

5 Save Sequence.

5.1 Under ***Sequence***, select ***Save Sequence***.

6 Print Sequence.

6.1 Under ***Sequence***, select ***Print Sequence***.

6.2 Check ***Method and Injection Info Part***; then click on ***Print***.

6.3 Verify that the sequence matches the vial placement on the autosampler.

6.4 Place sequence printout in the sequence binder.

7 Starting Sequence.

7.1 Under ***Run Control***, select ***Run Sequence***.

8 During the Sequence Run.

8.1 Print out the calibration curve for the standards.

8.1.1 Under ***View***, select ***Data Analysis***.

8.1.2 Under ***File***, select ***Print***.

8.1.3 Select ***Calib Table + Curves***.

8.2 Verify the correlation coefficient for each compound present in the calibration is greater than 0.98.

8.2.1 If the calibration fails, the sequence is stopped and corrective action is implemented.

8.3 Verify control and check values are within control limits.

9 After Sequence Completion.

9.1 Review the chromatograms. Verify the MPA peak is of a consistent area, throughout the entire sequence. If a sample was misinjected, the problem will most likely show up in the MPA peak.

10 Note any problems in the lab instrument notebook.

Appendix C

ANALYSIS FOR SILOXANES

Calibration Standard Stock. The stock standard consists of the following compounds, which are placed in a 500 mL volumetric flask and prepared, as follows:

Analyte	Amount (g)
Hexamethyldisiloxane	40
Hexamethylcyclotrisiloxane	40
Octamethyltrisiloxane	40
Octamethylcyclotetrasiloxane	40
Decamethyltetrasiloxane	40
Benzyl Alcohol	40
Decamethylcyclopentasiloxane	40

Bring to volume with MPA and mix. Aliquot the solution into 20 mL screw-capped vials. Date, label and store the vials in the Standards refrigerator.

Control/Check Stock: The control/check stock is a 20% hexamethyldisiloxane (HMDS) solution prepared by weighing 20 g of HMDS into a 100 mL volumetric flask. Bring to volume with MPA and mix. The HMDS is weighed out in the preparation of the stock, so the concentration is already g/mL. Store the control/check stock in individual 8 mL screw-capped vials in the Standards refrigerator.

1 Load Method.

1.1 Under ***Method***, select ***Load Method***.

1.2 Click on ***silox.m***; then ***OK***.

2 Load Sequence.

2.1 Under ***Sequence***, select ***Load Sequence***.

2.2 Click on ***SILOX.S***; then ***OK***.

3 Modifying Sequence.

3.1 Under ***Sequence***, select ***Sequence Parameters*** to create a subdirectory for the data.

3.2 Input sequence parameters.

3.2.1 **Operator Name** – enter your three initials.

3.2.2 **Data Files** – select ***Auto***.

3.2.3 **Subdirectory** – enter the two decimal month, day, year (December 5, 2007 would be entered as ***120507***); a letter can be put at the end to distinguish specific analyses run on the same day.

3.2.4 **Part of method to run** – select ***According to Runtime Checklist***.

3.2.5 **Sequence Comment** – enter set sample numbers, and any other pertinent information.

3.3 Click on ***OK***.

4 Sequence Table.

4.1 Under ***Sequence***, select ***Sequence Table***.

4.2 Select the ***Back*** injector.

4.3 Input sequence information.

4.3.1 **Sample Name** – enter the following, in order: MPA blank, the five standards, MPA blank, control, and the samples.

4.3.1.1 An entry for MPA blank followed by a check is made after every ten samples and again at the end of the run.

4.3.2 **Method Name** – select ***SILOX*** from the pull-down menu.

4.3.3 **Vial** – vial number corresponds to the position of the vial on the autosampler tray.

4.3.3.1 Each entry for the blank will reference the same blank vial.

4.3.3.2 Each entry for check will reference the control vial.

4.3.4 **Inj/Vial** – “1”.

4.3.5 **Sample Type** – select ***Sample*** from the pull down menu for all lines except

for the five calibration lines; for these select ***Calibration***.

4.3.6 For calibration lines only:

4.3.6.1 **Cal Level** – enter 1,2,3,4,5, respectively.

4.3.6.2 **Update RF** – select ***Replace*** from the pull down menu.

4.3.6.3 **Update RT** – select ***Average*** from the pull down menu.

4.4 Click on ***OK***.

5 Save Sequence.

5.1 Under ***Sequence***, select ***Save Sequence***.

6 Print Sequence.

6.1 Under ***Sequence***, select ***Print Sequence***.

6.2 Check ***Method and Injection Info Part***; then click on ***Print***.

6.3 Verify that the sequence matches the vial placement on the autosampler.

6.4 Place sequence printout in the sequence binder.

7 Starting Sequence.

7.1 Under ***Run Control***, select ***Run Sequence***.

8 During the Sequence Run.

8.1 Print out the calibration curve for the standards.

8.1.1 Under ***View***, select ***Data Analysis***.

8.1.2 Under ***File***, select ***Print***.

8.1.3 Select ***Calib Table + Curves***.

8.2 Verify the correlation coefficient for each compound present in the calibration is greater than 0.98.

8.2.1 If the calibration fails, the sequence is stopped and corrective action is implemented.

8.3 Verify control and check values are within control limits.

9 After Sequence Completion.

9.1 Review the chromatograms. Verify the MPA peak is of a consistent area, throughout the entire sequence. If a sample was misinjected, the problem will most likely show up in the MPA peak.

10 Note any problems in the lab instrument notebook.

Appendix D

ANALYSIS FOR METHYL ETHYL KETONE AND p-CHLOROBENZOTRIFLUORIDE

Calibration Standard Stock: The stock standard consists of the following compounds, which are placed in a 500 mL volumetric flask and prepared, as follows:

Analyte	Amount (g)
Methyl Ethyl Ketone	40
p-Chlorobenzotrifluoride	40
PM Acetate*	40
2-Butoxyethanol	40

*PM Acetate = Propylene Glycol Methyl Ether Acetate

Bring to volume with MPA and mix. Aliquot the solution into 20 mL screw-capped vials. Date, label and store the vials in the Standards refrigerator.

Control/Check Stock: The control/check stock is a 40% methyl ethyl ketone (MEK) solution prepared by weighing 40 g of MEK into a 100 mL volumetric flask. Bring to volume with MPA and mix. The MEK is weighed out in the preparation of the stock, so the concentration is already g/mL. Store the control/check stock in individual 8 mL screw-capped vials in the Standard refrigerator.

1 Load Method.

1.1 Under ***Method***, select ***Load Method***.

1.2 Click on ***mek.m***; then ***OK***.

2 Load Sequence.

2.1 Under ***Sequence***, select ***Load Sequence***.

2.2 Click on ***MEK.S***; then ***OK***.

3 Modifying Sequence.

3.1 Under ***Sequence***, select ***Sequence Parameters*** to create a subdirectory for the data.

3.2 Input sequence parameters.

3.2.1 **Operator Name** – enter your three initials.

3.2.2 **Data Files** – select ***Auto***.

3.2.3 **Subdirectory** – enter the two decimal month, day, year (December 5, 2007 would be entered as ***120507***); a letter can be put at the end to distinguish specific analyses run on the same day.

3.2.4 **Part of method to run** – select ***According to Runtime Checklist***.

3.2.5 **Sequence Comment** – enter set sample numbers, and any other pertinent information.

3.3 Click on ***OK***.

4 Sequence Table.

4.1 Under ***Sequence***, select ***Sequence Table***.

4.2 Select the ***Back*** injector.

4.3 Input sequence information.

4.3.1 **Sample Name** – enter the following, in order: MPA blank, the five standards, MPA blank, control, and the samples.

4.3.1.1 An entry for MPA blank followed by a check is made after every ten samples and again at the end of the run.

4.3.2 **Method Name** – select ***MEK*** from the pull-down menu.

4.3.3 **Vial** – vial number corresponds to the position of the vial on the autosampler tray.

4.3.3.1 Each entry for the blank will reference the same blank vial.

4.3.3.2 Each entry for check will reference the control vial.

4.3.4 **Inj/Vial** – “1”.

4.3.5 **Sample Type** – select ***Sample*** from the pull down menu for all lines except for the five calibration lines; for these select ***Calibration***.

4.3.6 For calibration lines only:

4.3.6.1 **Cal Level** – enter 1,2,3,4,5, respectively.

4.3.6.2 **Update RF** – select ***Replace*** from the pull down menu.

4.3.6.3 **Update RT** – select ***Average*** from the pull down menu.

4.4 Click on ***OK***.

5 Save Sequence.

5.1 Under ***Sequence***, select ***Save Sequence***.

6 Print Sequence.

6.1 Under ***Sequence***, select ***Print Sequence***.

6.2 Check ***Method and Injection Info Part***; then click on ***Print***.

6.3 Verify that the sequence matches the vial placement on the autosampler.

6.4 Place sequence printout in the sequence binder.

7 Starting Sequence.

7.1 Under ***Run Control***, select ***Run Sequence***.

8 During the Sequence Run.

8.1 Print out the calibration curve for the standards.

8.1.1 Under ***View***, select ***Data Analysis***.

8.1.2 Under ***File***, select ***Print***.

8.1.3 Select ***Calib Table + Curves***.

8.2 Verify the correlation coefficient for each compound present in the calibration is greater than 0.98.

8.2.1 If the calibration fails, the sequence is stopped and corrective action is implemented.

8.3 Verify control and check values are within control limits.

9 After Sequence Completion.

9.1 Review the chromatograms. Verify the MPA peak is of a consistent area, throughout the entire sequence. If a sample was misinjected, the problem will most likely show up in the MPA peak.

10 Note any problems in the lab instrument notebook.

Appendix E

ANALYSIS FOR GLYCOLS

Calibration Standard Stock: The stock standard consists of the following compounds, which are placed in a 500 mL volumetric flask and prepared, as follows:

Analyte	Amount (g)
Ethylene Glycol	40
Propylene Glycol	40
Carbitol	40
Glycerol	40
Butyl Carbitol	40
Triethylene Glycol	40

Bring to volume with MPA and mix. Aliquot the solution into 20 mL screw capped vials. Date, label and store the vials in the Standards refrigerator.

Control/Check Stock: The control/check stock is a 20% propylene glycol solution prepared by weighing 20 g of propylene glycol into a 100 mL volumetric flask. Bring to volume with MPA and mix. The propylene glycol is weighed out in the preparation of the stock, so the concentration is already g/mL. Store the control/check stock in individual 8 mL screw-capped vials in the Standard refrigerator.

1 Load Method.

1.1 Under ***Method***, select ***Load Method***.

1.2 Click on ***glycol.m***; then ***OK***.

2 Load Sequence.

2.1 Under ***Sequence***, select ***Load Sequence***.

2.2 Click on ***GLYCOL.S***; then ***OK***.

3 Modifying Sequence.

3.1 Under ***Sequence***, select ***Sequence Parameters*** to create a subdirectory for the data.

3.2 Input sequence parameters.

3.2.1 **Operator Name** – enter your three initials.

3.2.2 **Data Files** – select ***Auto***.

3.2.3 **Subdirectory** – enter the two decimal month, day, year (December 5, 2007 would be entered as ***120507***); a letter can be put at the end to distinguish specific analyses run on the same day.

3.2.4 **Part of method to run** – select ***According to Runtime Checklist***.

3.2.5 **Sequence Comment** – enter set sample numbers, and any other pertinent information.

3.3 Click on ***OK***.

4 Sequence Table.

4.1 Under ***Sequence***, select ***Sequence Table***.

4.2 Select the ***Back*** injector.

4.3 Input sequence information.

4.3.1 **Sample Name** – enter the following, in order: MPA blank, the five standards, MPA blank, control, and the samples.

4.3.1.1 An entry for MPA blank followed by a check is made after every ten samples and again at the end of the run.

4.3.2 **Method Name** – select ***GLYCOL*** from the pull-down menu.

4.3.3 **Vial** – vial number corresponds to the position of the vial on the autosampler tray.

4.3.3.1 Each entry for the blank will reference the same blank vial.

4.3.3.2 Each entry for check will reference the control vial.

4.3.4 **Inj/Vial** – “1”.

4.3.5 **Sample Type** – select ***Sample*** from the pull down menu for all lines except for the five calibration lines; for these select ***Calibration***.

4.3.6 For calibration lines only:

4.3.6.1 **Cal Level** – enter 1,2,3,4,5, respectively.

4.3.6.2 **Update RF** – select ***Replace*** from the pull down menu.

4.3.6.3 **Update RT** – select ***Average*** from the pull down menu.

4.4 Click on ***OK***.

5 Save Sequence.

5.1 Under ***Sequence***, select ***Save Sequence***.

6 Print Sequence.

6.1 Under ***Sequence***, select ***Print Sequence***.

6.2 Check ***Method and Injection Info Part***; then click on ***Print***.

6.3 Verify that the sequence matches the vial placement on the autosampler.

6.4 Place sequence printout in the sequence binder.

7 Starting Sequence.

7.1 Under ***Run Control***, select ***Run Sequence***.

8 During the Sequence Run.

8.1 Print out the calibration curve for the standards.

8.1.1 Under ***View***, select ***Data Analysis***.

8.1.2 Under ***File***, select ***Print***.

8.1.3 Select ***Calib Table + Curves***.

8.2 Verify the correlation coefficient for each compound present in the calibration is greater than 0.98.

8.2.1 If the calibration fails, the sequence is stopped and corrective action is implemented.

8.2.2 Ethylene glycol has a four-point calibration.

8.3 Verify control and check values are within control limits.

9 After Sequence Completion.

9.1 Review the chromatograms. Verify the MPA peak is of a consistent area, throughout the entire sequence. If a sample was misinjected, the problem will most likely show up in the MPA peak.

10 Note any problems in the lab instrument notebook.

SOP Revision History

DATE	VERSION	NOTES
March 10, 1998	1.1	Adjusted document font to Times New Roman 12. Inserted appendix B formerly a stand-alone document.
February 3, 1999	1.2	Addition of exempt compounds in the calibration files. This also includes modifications to Appendix A to include additional analyses for some less common exempts and aromatic hydrocarbons.
February 4, 2003	1.3	Inserted Appendix C the Siloxane procedure and Appendix D the MEK procedure. Renamed the DCM procedure Appendix B, and renamed the Acetone procedure Appendix A. Adjusted document font to Times New Roman 12. Renumbered to new section number.
April 3, 2003	1.4	Modified all Appendices to reflect calibration curve changes, and calibration standard preparation. Modified calibration range for MEK and Siloxane, now to include a high point of 80 and 50 percent respectively. Modified Acetone/Alcohol standard prep exception including ethanol and isopropanol. Current neat ethanol is denatured with methanol.
January 7, 2005	1.5	Inserted Appendix E, the Glycol procedure. Retitled to reflect the scope covered by SOP. Changed document font to Arial 12. Corrected revision enumeration.
January 18, 2005	1.6	Added Glycerol and Butyl Carbitol to the Glycol procedure (Appendix E).